

Europäisches Patentamt  
European Patent Office  
Office européen des brevets



(11) EP 0 969 117 A2

(12) EUROPEAN PATENT APPLICATION

(43) Date of publication:  
05.01.2000 Bulletin 2000/01

(51) Int. Cl.<sup>7</sup>: C23C 14/08, C23C 14/30,  
C23C 14/02, C23C 28/00

(21) Application number: 99303419.8

(22) Date of filing: 30.04.1999

(84) Designated Contracting States:  
AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU  
MC NL PT SE  
Designated Extension States:  
AL LT LV MK RO SI

(30) Priority: 01.07.1998 US 108201

(71) Applicant:  
GENERAL ELECTRIC COMPANY  
Schenectady, NY 12345 (US)

(72) Inventors:  
• Rigney, David Vincent  
Cincinnati, Ohio 45230 (US)

• Wortman, David John  
Hamilton, Ohio 45011 (US)  
• Rigney, Joseph David  
Milford, Ohio 45150 (US)  
• Maricocchi, Antonio Frank  
Loveland, Ohio 45140 (US)  
• Bruce, Robert William  
Loveland, Ohio 45140 (US)

(74) Representative:  
Goode, Ian Roy et al  
London Patent Operation  
General Electric International, Inc.  
Essex House  
12-13 Essex Street  
London WC2R 3AA (GB)

(54) Method of forming a thermal barrier coating system

(57) A method for producing a thermal barrier coating system on an article that will be subjected to a hostile environment. The thermal barrier coating system is composed of a metallic bond coat and a ceramic thermal barrier coating having a columnar grain structure. The method generally entails forming the bond coat on the surface of a component (12), and then grit blasting the bond coat with an abrasive media having a particle size of greater than 80 mesh. The component (12) is then supported within a coating chamber (10) containing an ingot (16) of the desired ceramic material. An

absolute pressure of greater than 0.010 mbar is established within the chamber (10) with an oxygen-containing gas having an oxygen partial pressure of at least 0.006 mbar. Thereafter, the ceramic material is vaporized with an electron beam such that the vapor deposits on the surface of the component (12) to form a layer of the ceramic material on the surface. According to the invention, the above process steps and parameters significantly improve spallation resistance of the coating system.

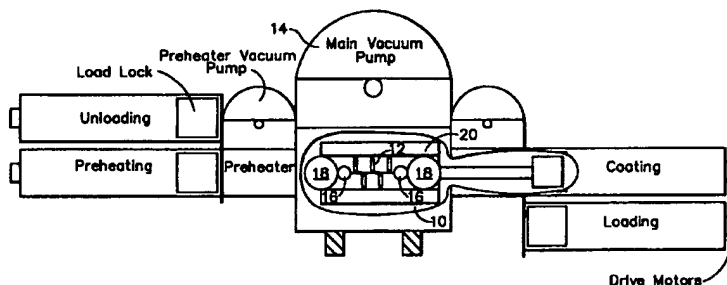


FIG. 1

EP 0 969 117 A2

## Description

[0001] This invention was made with Government support under Agreement No. N00019-92-C-0149 awarded by the U.S. Department of the Navy. The Government has certain rights in the invention.

5 [0002] This invention relates to thermal barrier coatings for components exposed to high temperatures, such as the hostile thermal environment of a gas turbine engine. More particularly, this invention is directed to a method for forming a thermal barrier coating system that exhibits improved resistance to spalling and low thermal conductivity.

[0003] Higher operating temperatures of gas turbine engines are continuously sought in order to increase their efficiency. However, as operating temperatures increase, the high temperature durability of the components of the engine  
10 must correspondingly increase. Significant advances in high temperature capabilities have been achieved through the formulation of nickel and cobalt-base superalloys, though such alloys alone are often inadequate to form components located in the hot sections of a gas turbine engine, such as the turbine, combustor and augmentor. A common solution is to thermally insulate such components in order to minimize their service temperatures. For this purpose, thermal barrier coatings (TBCs) formed on the exposed surfaces of high temperature components have found wide use.

15 [0004] To be effective, thermal barrier coatings must have low thermal conductivity, be capable of being strongly adhered to the article, and remain adherent through many heating and cooling cycles. The latter requirement is particularly demanding due to the different coefficients of thermal expansion between materials having low thermal conductivity and superalloy materials used to form turbine engine components. For this reason, thermal barrier coatings have generally employed a metallic bond coat deposited on the surface of a superalloy component. A thermal-insulating layer  
20 (the TBC) is then deposited on the bond coat, which together form what is termed a thermal barrier coating system. The metallic bond coat is typically a diffusion aluminide or an oxidation-resistant alloy, such as  $M\text{CrAlY}$  where M is iron, cobalt and/or nickel, which promotes the adhesion of the insulating layer to the component while also inhibiting oxidation of the underlying superalloy.

[0005] Various ceramic materials have been employed as the TBC, particularly zirconia ( $\text{ZrO}_2$ ) stabilized by yttria ( $\text{Y}_2\text{O}_3$ , magnesia ( $\text{MgO}$ ) or other oxides. These particular materials are widely employed in the art because they can be readily deposited by plasma spray, flame spray and vapor deposition techniques. A continuing challenge of thermal barrier coating systems has been the formation of a more adherent ceramic layer that is less susceptible to spalling when subjected to thermal cycling. For this purpose, the prior art has proposed various coating systems, with considerable emphasis on ceramic layers having enhanced strain tolerance as a result of the presence of porosity, microcracks and segmentation of the ceramic layer. Segmentation indicates that the ceramic layer has columnar grain boundaries or cracks oriented perpendicular to the surface of the component, and is achieved with electron beam physical vapor deposition (EBPVD) techniques. As is known in the art, a thermal barrier coating having a columnar grain structure is able to expand with its underlying substrate without causing damaging stresses that lead to spallation, as evidenced by thermal cyclic testing.

35 [0006] Zirconia-based thermal barrier coatings, and particularly yttria-stabilized zirconia (YSZ) coatings having columnar grain structures and a thickness on the order of about 125 micrometers (about 0.005 inch) or more are widely employed in the art for their desirable thermal and adhesion characteristics. Processes for producing these coatings with EBPVD techniques generally entail suspending a component in a coating chamber maintained at a pressure of 0.005 mbar or less. While greater pressures have been reported, such as in U.S. Patent Nos. 4,006,268 and 5,716,720,  
40 the 0.005 mbar upper pressure limit is adhered to those skilled in the art when depositing thermal barrier coatings because of the operational characteristics of electron beam guns and the baffling chambers. For example, higher pressures are avoided because control of the electron beam is more difficult at pressures above about 0.005 mbar, with erratic operation being reported at coating chamber pressures above 0.010 mbar. It has also been believed that the life of the gun filament would be reduced or the gun contaminated if operated in pressures above 0.005 mbar.

45 [0007] Though YSZ deposited by EBPVD is a highly successful coating system for protecting turbine engine components, there is an ongoing effort to improve the spallation resistance and further reduce the thermal conductivity of thermal barrier coatings. Accordingly, it would be desirable if thermal barrier coating systems that exhibit improved spallation resistance and reduced thermal conductivity could be produced with conventional materials by improving the processes by which such coating systems are deposited.

50 [0008] The present invention is a method for producing a thermal barrier coating system on an article which will be subjected to a hostile environment that promotes spallation, as is the case with turbine, combustor and augmentor components of a gas turbine engine. The thermal barrier coating system is composed of a metallic bond coat and a ceramic thermal barrier coating, such as yttria-stabilized zirconia (YSZ) having a columnar grain structure. According to the invention, the bond coat is prepared and the ceramic subsequently deposited by electron beam physical vapor deposition (EBPVD)-using process parameters that significantly improve spallation resistance and reduce the thermal conductivity of the ceramic thermal barrier coating.

55 [0009] The method of this invention generally entails forming a bond coat on a surface of a component, such as an airfoil, and then grit blasting the bond coat with an abrasive media having a particle size of about 250 to about 300  $\mu\text{m}$

(e.g., about 54 mesh) and at a pressure of greater than 60 psi. The component is then supported within a coating chamber containing an ingot of ceramic material. Most critical to this invention is that an absolute pressure of greater than 0.010 mbar is established within the coating chamber with an oxygen-containing gas, after which the ceramic material is melted and vaporized with an electron beam such that the vapor deposits on the surface of the component to form a layer of the ceramic material on the surface.

[0010] According to the invention, a preferred pressure within the coating chamber is about 0.012 to 0.015 mbar, and the oxygen-containing gas has an oxygen partial pressure of at least 0.006 mbar (50%). Finally, the ceramic material is preferably evaporated at a rate of about 23.5 to about 29.2 grams per minute for each source (e.g., ingot) of ceramic - according to this invention. It is the evaporation rate of each ceramic source that is critical, and not the combined evaporation rate of all sources. Surprisingly, under these conditions the ceramic coating material is deposited to form a porous thermal barrier layer that tenaciously adheres to the bond coat. Even more surprisingly, the ceramic coating is significantly more spall resistant than thermal barrier coatings deposited under conventional conditions, i.e., lower chamber pressures, coarser abrasive grit, lower oxygen partial pressures and/or lower evaporation rates than those employed by the method of this invention, with chamber pressure appearing to be the most critical parameter to the present invention. Also unexpected is that the coating deposition rate is dependent on chamber pressure, with higher pressures corresponding to higher deposition rates and more efficient usage of ceramic material, i.e., more ceramic deposited on a turbine component per unit length of ingot consumed (volume evaporated). Accordingly, the method of the present invention not only improves the spallation resistance of the resulting TBC, but also improves manufacturing economies.

[0011] Other objects and advantages of this invention will be better appreciated from the following detailed description.

[0012] The above and other advantages of this invention will become more apparent from the following description taken in conjunction with the accompanying drawings, in which:

Figure 1 shows a schematic representation of an electron beam physical vapor deposition apparatus used to deposit a ceramic layer of a thermal barrier coating system in accordance with this invention;

Figure 2 is a graph indicating a correlation between TBC thickness and coating chamber pressure and ingot feed (evaporation) rate; and

Figure 3 is a graph indicating a strong correlation between %O<sub>2</sub> of total pressure on FCT life in relation to ingot feed (evaporation) rate.

[0013] The present invention is generally directed to a method for depositing a thermal barrier coating on a metal component intended for operation within an environment characterized by high temperatures. Notable examples of such components include the high and low pressure turbine nozzles and blades, shrouds, combustor liners and augmentor hardware of gas turbine engines. While the advantages of this invention will be described with reference to turbine blades of gas turbine engines, the teachings of this invention are generally applicable to any component in which a thermal barrier coating can be used to insulate the component from a hostile thermal environment.

[0014] To illustrate the invention, the coating chamber 10 of an electron beam physical vapor deposition (EBPVD) coating apparatus is shown in Figure 1. Shown supported within the coating chamber 10 are turbine blades 12 of a gas turbine engine. Coating chambers of the type shown in the Figure are typically capable of being maintained at a vacuum level of about  $1 \times 10^{-3}$  Torr (about 0.001 mbar) or less. A vacuum pump 14 of a type known in the art is employed to evacuate the chamber 10 during the deposition process.

[0015] As is generally conventional, the blades 12 may be formed of a nickel-base or cobalt-base superalloy. Each blade 12 includes an airfoil section against which hot combustion gases are directed during operation of the gas turbine engine, and whose surface is therefore subjected to hostile thermal and chemical environment. For this reason, a thermal barrier coating system is adherently formed on the surface of the blade 12 to protect its superalloy substrate. As is generally typical in the art, the coating system is composed of a metallic bond coat over which a ceramic coating is formed. The bond coat is preferably an oxidation-resistant metal composition that protects the underlying superalloy substrate from oxidation and enables the ceramic coating to more tenaciously adhere to the substrate. Suitable bond coat materials include diffusion aluminide containing intermetallic platinum and/or nickel aluminide phases, and MCrAlY alloys such as NiCrAlY. The thickness for a diffusion aluminide bond coat is at least about 25 micrometers, with a suitable range being about 50 to 75 micrometers. A typical thickness range for MCrAlY bond coats is about 25 to 375 micrometers.

[0016] Following deposition of the bond coat, the surface of the bond coat is prepared for deposition of the ceramic coating by abrasive grit blasting. In the past, grit blasting with 80 mesh or finer alumina particles has been preferred as the abrasive medium to clean the bond coat surface and produce the smooth surface finish required for narrow, parallel columnar PVD ceramic coatings. However, according to this invention, a coarser abrasive medium has been unexpectedly found to promote the adhesion of the ceramic coating to the bond coat. A particle size of about 250 to about 300

micrometers is suitable, with a preferred particle size being about 54 mesh (about 275 $\mu$ m). The abrasive medium is preferably discharged under a pressure of about 60 psi or more at a standoff distance of about 7 to about 10 inches (about 17.5 to about 25.4 centimeters) from a standard unit used for this purpose. Thereafter, an alumina scale is formed on the bond coat at an elevated temperature to promote adhesion of the ceramic coating. The alumina scale, often referred to as a thermally grown oxide or TGO, develops from oxidation of the aluminum-containing bond coat either through exposure to elevated temperatures during deposition of the ceramic coating, or by way of a high temperature treatment specifically performed for this purpose.

[0017] Finally, the ceramic coating is deposited by EBPVD under conditions that were unexpectedly determined to have a significant effect on the spall resistance and thermal conductivity of the coating. As shown in Figure 1 and well known in the art, the ceramic coating is deposited from a vapor formed by melting and vaporizing one or more ingots 16 with an appropriate number electron beams from electron beam gun 18. A preferred ceramic coating material is zirconia (ZrO<sub>2</sub>) partially or fully stabilized by yttria (e.g., 3%-20%, preferably 4%-8% Y<sub>2</sub>O<sub>3</sub>), though yttria stabilized with magnesia, ceria, calcia, scandia or other oxides could be used. The blades 12 are preferably secured to a rotatable support 20 of a type known in the art, and the ingots 16 are located within the chamber 10 such that their upper ends are adjacent the blades 12. The electron beams emitted by the guns 18 are directed toward the upper ends of the ingots 16, which causes the surfaces of the ingots 16 to melt and form molten pools of the ceramic material. Intense heating of the ceramic material by the electron beams causes molecules of the ceramic material to evaporate, travel upwardly, and then deposit on the surfaces of the blades 12. A heater (not shown) may be positioned above the blades 12 to provide additional heating as may be required to supplement the heat provided by the vaporized ceramic material and the radiation from the molten surface of the ingots 16. In this manner, the blades 12 are brought to a suitable deposition temperature of about 925°C to about 1140°C. A stable blade temperature promotes the desired columnar grain structure for the ceramic layer in which the longitudinal axis of each grain is roughly perpendicular to the surface of each blade 12.

[0018] In the prior art, a vacuum of at most 0.010 mbar, and more typically about 0.005 mbar, would be drawn within the chamber 10 to perform the coating process, the reason being that higher pressures were known to cause erratic operation of the electron beam guns 18 and make the electron beams difficult to control, with the presumption that inferior coatings would result. However, according to this invention, coating chambers such as that shown in Figure 1 are operated at higher pressures that surprisingly yield a ceramic coating with improved spallation and impact resistance, as well as promote the coating deposition rate in conjunction with higher ingot evaporation rates than that achieved in the prior art. Furthermore, prior art practice is to perform the coating process by flowing oxygen gas into the coating chamber 10. However, according to this invention, flowing an oxygen-argon mixture to achieve an oxygen partial pressure above 50% (e.g., about 0.006 mbar) has also been determined to promote the spallation resistance of the resulting ceramic coating.

[0019] During a preliminary investigation leading to the present invention, conventional (baseline) coating parameters were evaluated with test specimens formed of René N5 superalloy (nominal composition in weight percent: 7.5Co, 7.0Cr, 1.5Mo, 5.0W, 3.0Re, 6.5Ta, 6.2Al, 0.15Hf, 0.05C, 0.004B, 0.01Y, balance Ni). PtAl bond coats were formed on the specimens by platinum plating followed by vapor phase aluminizing, and their surfaces prepared with either 36, 54 or 80 mesh alumina abrasive media discharged at pressures of 40, 70 and 60 psi, respectively (about 0.28, 0.41 and 0.48 MPa, respectively). The different discharge pressures served to achieve a similar surface roughness or texture (about 50 to 60  $\mu$ inches Ra) for each of the specimens. The specimens were then coated with YSZ by EBPVD using the following parameters: coating temperature of about 1000°C, coating pressure of about 0.012 mbar, and an ingot feed rate of about 2.1 mm/minute using two ingots having diameters of about 6.35 cm (yielding an evaporation rate of about 24.6 grams/minute per ingot, for a combined evaporation rate of about 49.2 g/minute). Furnace cycle testing (FCT) was then performed on the specimens at 2075°F (about 1135°C) with one-hour cycles, with specimens being removed from test once 10% of the coating surface area had spalled. On average, the specimens whose bond coats were surface treated with 36 mesh abrasive media exhibited an average FCT life of 493 cycles, while those treated with 54 and 80 mesh media exhibited FCT lives of about 593 and 540 cycles, respectively.

[0020] Based on the above promising results, a second investigation was then pursued to evaluate various coating parameters with test specimens formed of Inconel 601 superalloy (nominal composition in weight percent: 23Cr, 1.4Al, 0.5Mn, 0.05C, 0.25Si, balance Ni). The coating parameters included preheat temperatures of about 900°C to about 1100°C, coating temperatures of about 1000°C to about 1150°C, component rotational speeds of about 6 to about 60 rpm, ingot feed rates of about 1.3 to about 2.5 mm/minute for each of two ingots with diameters of about 6.35 cm (equivalent to an evaporation rate of about 15.2 to about 29.2 grams per minute per ingot, for a combined ingot evaporation rate of about 30.3 to about 58.5 g/minute), and coating chamber pressures of about 0.001 to about 0.0195 mbar. All specimens were provided with a diffusion aluminide bond coat having a thickness in the range of about 25 to about 75 micrometers. The bond coats were surface prepared with 80 mesh alumina abrasive media discharged at a pressure of about 60 psi (about 0.41 MPa).

[0021] Deposition of the ceramic coating on each specimen was performed in two coating chambers similar to that

shown in Figure 1. The specimens were preheated to about 900°C to about 1100°C, after which the coating process brought the specimens to the coating temperatures indicated above. As was expected, the operation of the guns significantly changed over the range of pressures tested. At the lower pressures (e.g., 0.001 mbar), the focus effects were more gradual and easier to control, and it was observed that high evaporation rates could be maintained without "spitting" of the molten ceramic pool formed at the upper end of the ingots. At pressures of 0.015 and 0.0195 mbar, the focus changes were much more critical, with small focus changes producing significant changes in evaporation rate. High ingot feed rates (e.g., 2.5 mm/minute per ingot, equivalent to a combined evaporation rate of 58.5 g/minute when using two ingots) required sharper focus adjustments, and the melt became more prone to spits and violent gas eruptions. Feed rates of 2.1 mm/minute per ingot (about 24.6 g/minute per ingot) reduced spitting to a level acceptable for the experiment.

[0022] The results of FCT testing performed on specimens processed with the above coating parameters are summarized below in Table I. Preheat temperature and rotational speeds are not indicated in Table I - generally, preheat temperatures of 1100°C had a positive effect, while rotational speeds did not appear to have any effect on coating results.

TABLE I

Chamber Pressure (mbar)	Coating Temp. (°C)	Ingot Feed Rate (mm/min.)	Total Feed Rate (g/min.)	Coating Thickness per mm ingot used (μm/mm)
0.000959	1013	1.32	30.9	2.92
0.000975	1112	2.02	47.3	4.57
0.007815	1054	2.25	52.6	4.45
0.007877	1137	2.27	53.1	3.91
0.007898	1090	2.10	49.2	4.60
0.01423	1032	1.33	31.1	4.85
0.0143	1099	1.33	31.1	6.53
0.0195	1138	2.28	53.4	6.99

The above results evidenced that deposition efficiencies (indicated by the amount of coating deposited per unit length of ingot consumed) substantially increased with higher coating chamber pressures. Such a result was unexpected, and was the basis for initiating further investigation into the effects of using deposition chamber pressures above that conventionally used in the art, e.g., above 0.005 mbar.

[0023] Subsequent testing was performed on test specimens formed of Inconel 601 and René N5 superalloys. A preheat temperature of 1100°C and a coating rotational speed of 6 rpm were employed throughout this phase of tests. Test parameters evaluated were coating temperatures of 1000 to 1150°C, ingot feed rates of about 1.3, 1.7 or 2.1 mm/minute using two ingots with diameters of about 6.35 cm (an evaporation rate of about 15.2, 19.9 or 24.6 g/minute per ingot, for a combined evaporation rate of about 30.3, 39.8 or 49.2 g/minute), partial pressures of 0% to 100% (the balance argon), and coating chamber pressures of about 0.001 to about 0.015 mbar. All René N5 specimens were provided with a platinum aluminide (PtAl) bond coat having a thickness of about 1.3 to 3 mils (about 33 to 76 micrometers), while the Inconel 601 specimens were provided with a diffusion aluminide bond coat having a thickness of about 0.9 to 2.5 mils (about 23 to 64 micrometers). The bond coats were surface prepared with either 54 mesh alumina abrasive media discharged at a pressure of about 70 psi (about 0.48 MPa) or 80 mesh alumina abrasive media discharged at about 60 psi (about 0.41 MPa), again the different pressures serving to achieve similar surface roughnesses or textures for all specimens. The specimens were then vacuum heat treated for two hours at about 1950°F (about 1066°C).

[0024] The coating process was controlled such that an equal mass of ingot was evaporated in each experiment. During coating, no significant difference in "spitting" was observed at the different parameters. Following deposition of the ceramic coating, spallation resulting from process causes was observed with four specimens, three of which were coated at pressures of 0.001 mbar and the fourth was coated at a low temperature (about 950°C) with no oxygen flowing in the system. Some of the remaining specimens were examined for coating thickness, while others underwent furnace cycle testing at about 2075°F (about 1135°C) with one-hour cycles.

[0025] The average coating thickness for all specimens was about 125 micrometers. Those specimens whose bond coats were surface treated with 54 mesh abrasive had an average coating thickness of 1.9% greater than the overall

average, and those whose bond coats were surface treated with 80 mesh abrasive had an average coating thickness of 2.7% less than the overall average. Since all specimens were exposed to the same coating conditions, these differences in coating thickness translated to differences in porosity within the coating, which was unexpected but beneficial to reducing internal coating stress and lowering thermal conductivity. Coating temperature and oxygen partial pressure did not significantly effect coating thickness. In contrast, a marked correlation was made when compared on the basis of total coating chamber pressure and ingot feed (evaporation) rate. Increased total coating chamber pressure (from 0.001 to 0.015 mbar) increased coating thickness, while increased feed rates (from 1.3 to 2.1 mm/minute per ingot, or 15.2 to 24.6 g/minute per ingot) resulted in lower deposition rates per quantity of ingot used, regardless of chamber pressure. These relationships are represented in Figure 2, and evidence an interaction that has a substantial economic effect of the EBPVD process itself. Specifically, deposition times can be reduced and deposition efficiencies (amount of ceramic deposited per unit of ingot consumed) improved with higher coating pressures.

[0026] Metallographic examinations of the specimens showed that all ceramic coatings had columnar microstructures, as would be expected from the EBPVD process used. However, the particular structure of the grains appeared to be dependent on the coating pressure. Fern-like or feather-like columnar grains were more often associated with coatings deposited at high total coating chamber pressures (0.015 mbar), while spike-like and branched columnar grains were more likely to be observed for coatings deposited at higher oxygen partial pressure levels (100% oxygen flow).

[0027] The FCT schedule entailed the completion of twenty one-hour cycles per day over a period of about three months with over 200 specimens. A specimen was removed from test once 10% of the coating surface area had spalled. The results of the FCT test are tabulated in Table II below.

TABLE II

Chamber Pressure (mbar)	Coating Temp. (°C)	Ingot Feed Rate (mm/min.)	O <sub>2</sub> Part. Press. (%)	FCT Life	
				54 Mesh (cycles)	80 Mesh (cycles)
0.001	1000	1.3	0	225	420
0.001	1000	2.1	0	493	420
0.001	1075	1.7	50	593	487
0.001	1150	1.3	100	480	506
0.001	1150	2.1	100	646	520
0.008	1000	1.7	50	500	493
0.008	1075	1.3	50	753	446
0.008	1075	1.7	0	480	333
0.008	1075	1.7	50	686	566
0.008	1075	1.7	50	526	420
0.008	1075	1.7	100	486	533
0.008	1075	2.1	50	700	386
0.008	1150	1.7	50	693	573
0.015	1000	1.3	100	660	446
0.015	1000	2.1	100	673	466
0.015	1075	1.7	50	646	440
0.015	1150	1.3	0	433	406
0.015	1150	2.1	0	560	486

[0028] The above results evidence a strong correlation between total coating chamber pressure, evaporation rate, oxygen partial pressure and grit media used with FCT life. When analyzed on the basis of bond coat surface treatment, those specimens treated with the 54 mesh media exhibited an average FCT life of 569 cycles, as compared to 463 cycles for the 80 mesh specimens. Eliminating the results obtained at 0.001 mbar pressure and "0%" oxygen conditions

evidenced an even more consistent correlation between FCT life and bond coat surface preparation, yielding average FCT lives for the 54 and 80 mesh specimens of 632 and 477 cycles, respectively. Under this analysis, the 54 mesh grit surface preparation technique exhibited an approximately 32% FCT life improvement over the 80 mesh specimens. Furthermore, with "0%" oxygen specimens eliminated, the average FCT life for specimens grit blasted with 54 mesh media and coated at 0.015 mbar was 660 cycles, as compared to an average life of 573 for specimens coated at 0.001 bar (an improvement of about 15%). Finally, Figure 3 shows the strong influence that oxygen partial pressure had on FCT life in relation to ingot feed rates between 1.3 and 2.1 mm/minute (evaporation rates of 15.2 to 24.6 g/minute per ingot, for a combined evaporation rate of 30.3 to 49.2 g/minute).

[0029] Statistical analysis of the results verified that the most important factors determining FCT life (2075°F, one-hour cycles) were oxygen partial pressure, ingot evaporation rate and total coating chamber pressure, with pressure being the greatest single factor. Analysis of the FCT life data on the basis of these parameters gave the following regression:

$$\text{FCT life} = 1309.7 + 30236(P) - 1407(R) + 8.15(O) - 13318(P)(R) + 484(R)^2 - 0.066(O)^2$$

where:

"FCT life" is in hours;

"P" is total coating chamber pressure in mbars;

"R" is ingot evaporation rate in mm/minute for each 6.35cm diameter ingot (ceramic source); and

"O" is oxygen partial pressure in percent of total coating chamber pressure.

[0030] Goodness of fit ( $R^2$ ) for this equation was 74%, and  $R^2$  adjusted was 60%. This analysis predicts that, using a preferred evaporation rate of 6.35 to 7.9 cm<sup>3</sup>/minute per ingot (equivalent to an evaporation rate of about 23.5 to 29.2 grams/minute per ingot, for a combined evaporation rate of about 47.0 to 58.5 g/minute), parameters for FCT life are an oxygen partial pressure of about 60%, more precisely about 61.4%, and a total coating chamber pressure of about 0.012 mbar, more precisely about 0.0117 mbar, and would yield an average (Xbar) FCT life of about 878 cycles.

[0031] The effect that the coating process parameters of this invention have on coating erosion and impact resistance was also evaluated. In service, thermal barrier coatings on gas turbine engine components are required to withstand damage from impact by hard particles of varying sizes that enter the high velocity gas stream through the air intake of a gas turbine engine, or are generated upstream in the engine. The result of impingement can be erosive wear (generally from smaller particles) or impact spallation from larger particles.

[0032] A test developed to measure the relative performance of coatings at high temperatures (2250°F, 1232°C) and high gas velocities (Mach 0.3) was modified to allow the injection of an alumina powder to evaluate erosion and impact resistance. Alumina powders with average particle sizes of about 50  $\mu\text{m}$  and about 560  $\mu\text{m}$  were used to evaluate erosion and impact resistance, respectively. Pin specimens of 0.25 inch diameter (about 6.35 mm) were formed of René N5 and then coated with a PtAl bond coat and a five mil (125  $\mu\text{m}$ ) thick EBPVD thermal barrier coating. The specimens were rotated at a rate of about 500 rpm during the experiment. Erosion resistance was measured in terms of the number of grams of erodent required to break through the thermal barrier coating to the underlying bond coat.

[0033] The thermal barrier coatings were processed in coating chambers maintained at an absolute pressure of either 0.006 or 0.012 mbar, with a feed rate of either about 1.7 or 2.1 mm/minute per ingot (equivalent to an evaporation rate of 20 or 24.6 g/minute per ingot, or a combined evaporation rate of 40 or 49.2 g/minute), and an oxygen partial pressure of either about 100% or 50%. Under erosion conditions, thermal barrier coatings deposited at 0.012 mbar exhibited about 10% greater erosion resistance than those deposited at 0.006 mbar. Under impact conditions, the thermal barrier coatings deposited at 0.012 mbar exhibited average spallation lives of about 1.3 times that of the lower pressure specimens. Accordingly, these experiments evidenced that thermal barrier coatings formed at pressures within the scope of this invention exhibit superior erosion and impact resistance as compared to coatings deposited at conventional coating pressures.

[0034] Finally, the effect that the coating process parameters of this invention have on thermal conductivity was also evaluated. Test specimens having dimensions of about 0.5 x 0.5 x 0.025 inch (12.7 x 12.7 x 0.635 mm) were machined from a René N5 substrate, surface treated to achieve a surface roughness of about 60  $\mu\text{m}$  Ra, and then coated with a five mil (125  $\mu\text{m}$ ) thick EBPVD thermal barrier coating. Thermal conductivity was calculated from the product of coating density, thermal diffusivity and specific heat. Coating density and thermal diffusivity are functions of coating microstructure (e.g., porosity level) while specific heat is mainly composition-dependent. Bulk coating density was calculated from weight gain and volume. Specific heat and thermal diffusivity were measured using differential scanning calorimetry and laser flash techniques, respectively, both of which were performed in accordance with ASTM standards.

[0035] The thermal barrier coatings were processed in coating chambers maintained at a feed rate of about 1.7 or 2.1 mm/minute per ingot (an evaporation rate of about 20 to 24.6 g/minute per ingot, or a combined evaporation rate of 40

or 49.2 g/minute), an absolute pressure of either 0.006 or 0.015 mbar, and an oxygen partial pressure of either about 100% or 50%, respectively. The average thermal conductivity values at 1100°C were 1.284 and 1.036 W/mK for the 0.006 and 0.015 mbar specimens, respectively, evidencing that coatings deposited at pressures within the scope of this invention exhibit significantly lower thermal conductivity as compared to coatings deposited at conventional coating pressures. Consequently, thermal barrier coatings formed in accordance with this invention are able to provide better thermal insulation for the components on which they are deposited.

#### Claims

1. A method for forming a ceramic layer on a component, the method comprising the steps of:

establishing an absolute pressure of greater than 0.010 mbar within a coating chamber (10) with an oxygen-containing gas; and

evaporating a ceramic material with an electron beam focused on at least one mass (16) of the ceramic material so as to produce a vapor of the ceramic material, the vapor depositing on a surface of a component (12) within the coating chamber (10) to form a layer of the ceramic material on the surface.

2. A method as recited in claim 1, further comprising the step of forming a bond coat on the surface of the component (12) prior to forming the layer of ceramic material on the surface of the component (12).

3. A method as recited in claim 1, further comprising the step of grit blasting the surface of the component (12) with an abrasive media having a particle size of about 250 to about 300  $\mu\text{m}$  and at a pressure of greater than 60 psi.

4. A method as recited in claim 1, wherein the ceramic material is yttria-stabilized zirconia.

5. A method as recited in claim 1, wherein the absolute pressure within the coating chamber (10) is at least 0.012 mbar.

6. A method as recited in claim 1, wherein the oxygen-containing gas has an oxygen partial pressure of at least 0.006 mbar.

7. A method as recited in claim 1, wherein the surface of the component (12) is at a temperature of about 925°C to about 1140°C as the vapor is deposited thereon during the evaporating step.

8. A method as recited in claim 1, wherein the ceramic material is evaporated at a rate of about 23.5 to about 29.2 grams/minute per mass (16).

9. A method as recited in claim 1, further including the step of preheating the surface of the component (12) to a temperature of about 1100°C prior to depositing the layer of ceramic material on the surface of the component (12).

10. A method for forming a thermal barrier coating on a component, the method comprising the steps of:

forming a bond coat on a surface of the component;

grit blasting the surface of the component with an abrasive media;

supporting the component within a coating chamber containing at least one ingot of yttria-stabilized zirconia; establishing an absolute pressure of greater than 0.010 mbar within the coating chamber with a gas having an oxygen partial pressure of at least 50%; and evaporating each ingot at a rate of about 23.5 to about 29.2 grams per minute with an electron beam focused on each ingot so as to produce a vapor of yttria-stabilized zirconia, the vapor depositing on the surface of the component to form a layer of yttria-stabilized zirconia on the surface.



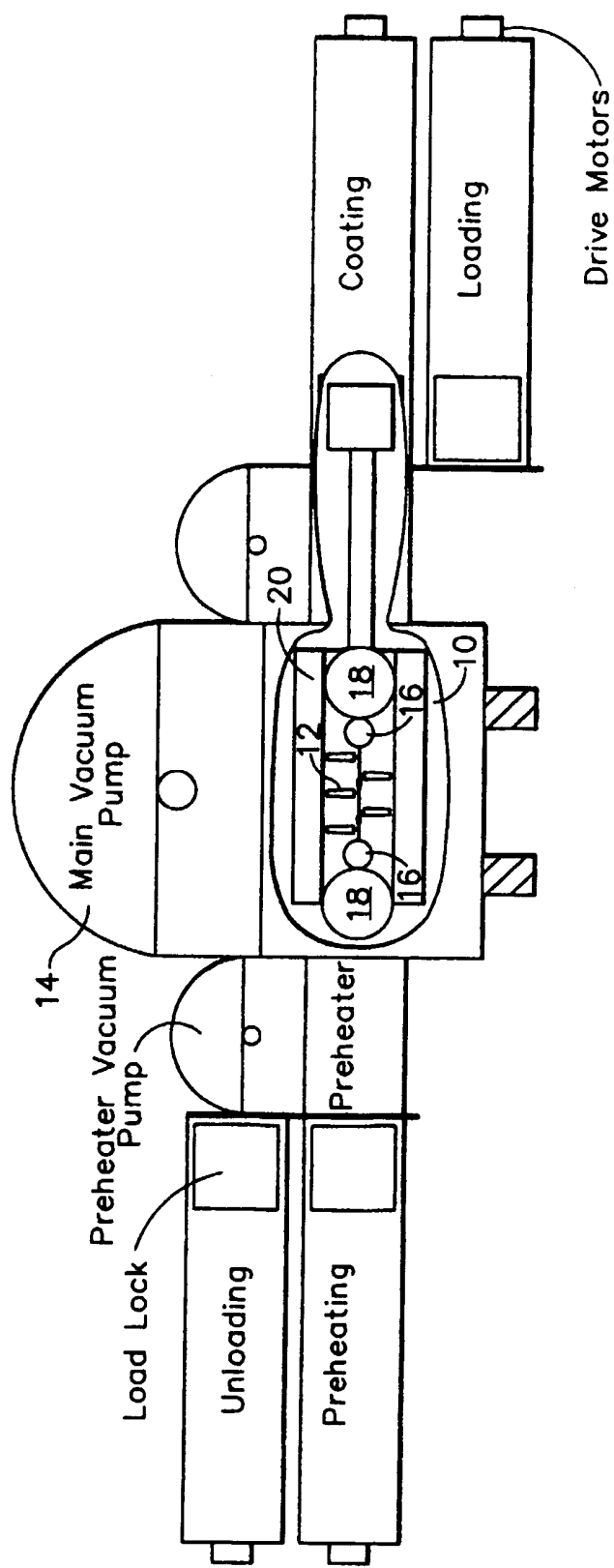


FIG. 1

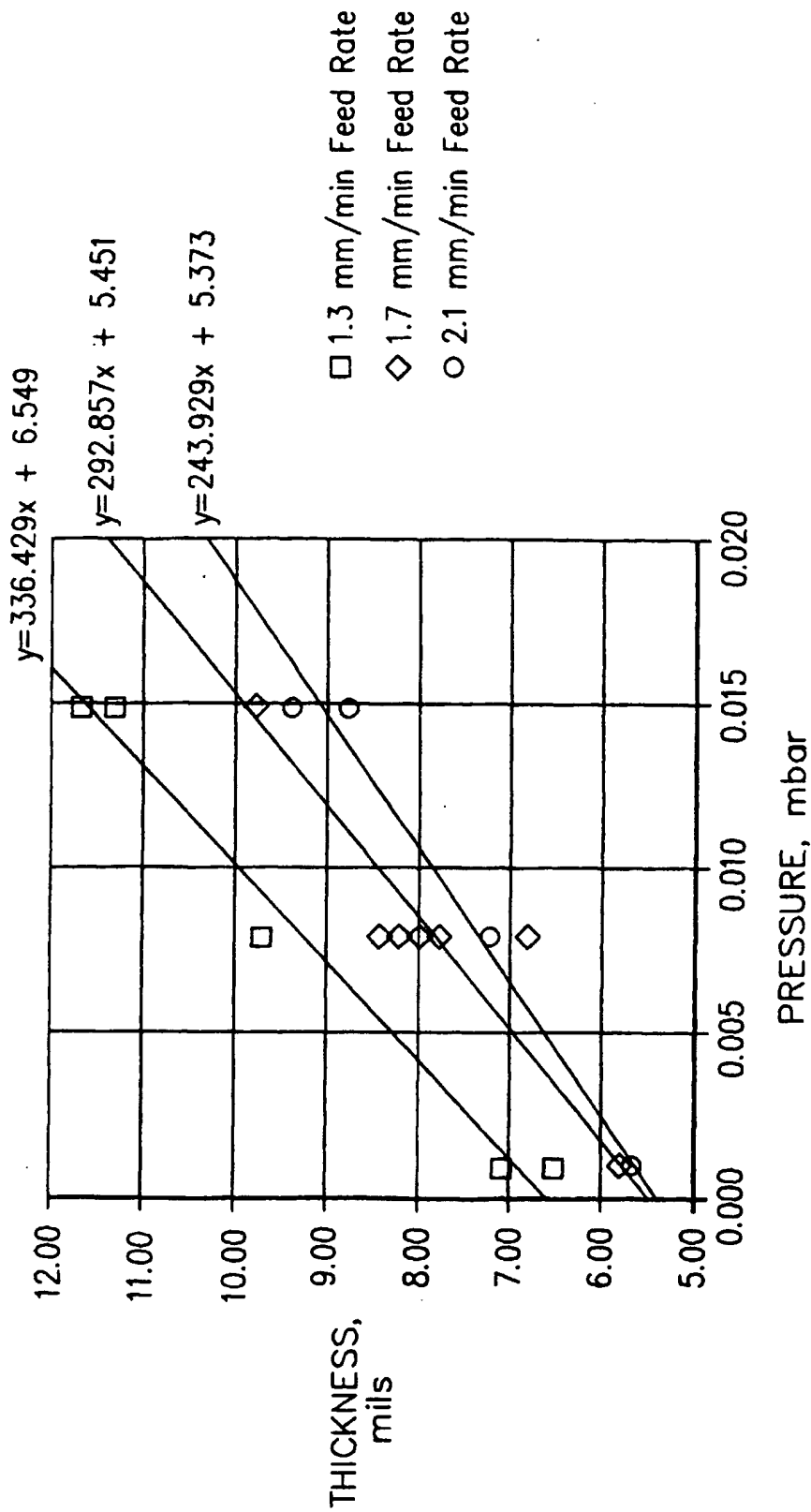


FIG. 2

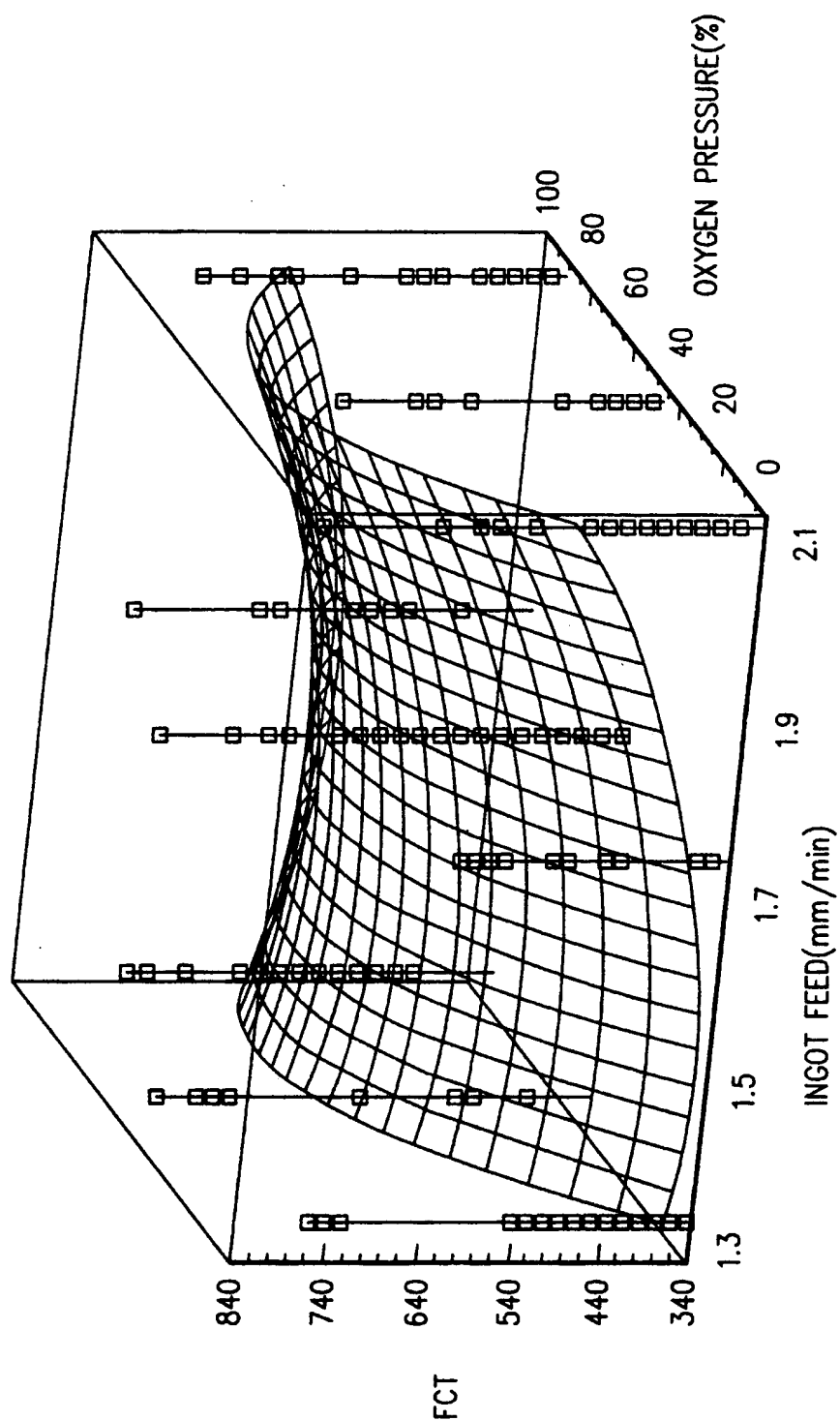


FIG. 3